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Search History

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90 L30

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<u>L27</u>	latex or L26	393629	<u>L27</u>
<u>L26</u>	rubber or rubbery or elastomer	367960	<u>L26</u>
<u>L25</u>	l23 and L24	97	<u>L25</u>
<u>L24</u>	l11 or l18	12035	<u>L24</u>
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'5274035'| '5665820'| '5112909'| '6013726'| '5128410'| '6103829'|
'4921909'| '5433984'| '5290876'| '4628072'| '5302665'| '5686528')[PN]

END OF SEARCH HISTORY

PATENT ABSTRACTS OF JAPAN

(11)Publication number : **08-134298**
(43)Date of publication of application : **28.05.1990**

(51)Int.CI. C08L 25/12
C08J 3/20
C08L 55/02

(21)Application number : 06-273245 (71)Applicant : TORAY IND INC
(22)Date of filing : 08.11.1994 (72)Inventor : KISHIMOTO AKIHIKO
GOTO EIZO
KADOI AKIRA
NAKAGAWA KEIJI

(54) PREPARATION OF IMPACT-RESISTANT RESIN COMPOSITION

(57) Abstract

(57) Abstract:
PURPOSE: To prepare an impact-resistant resin compsn. which can reduce the necessity of waste water treatment and has excellent color tone and mechanical strengths by continuously adding and mixing a particular graft copolymer to a copolymer, in a melted state, prepnd by continuous bulk polymn. of a predetermined monomer mixture.

CONSTITUTION: 90 to 5 pts.wt. (hereinafter referred to as 'pts.') graft copolymer prep'd. by graft-polymerizing 95 to 20 pts. mixture of 10 to 100wt.% (hereinafter referred to as '%') arom. vinyl monomer (A), 0 to 50% of vinyl cyanide monomer (B), and 0 to 80% (meth)acrylic ester monomer (C), and (D) 0 to 60% other copolymerizable vinyl monomer(s) in the presence of 5 to 80 pts. rubber-like polymer and feeding a slurry or a water-contg. cake prep'd. from the resultant latex into an extruder provided with a groove, a hole, or a gap and a vent hole for passing a liquid material therethrough, thereby conducting dehydrating and drying, is continuously added to and mixed with 10 to 95 pts. copolymer, in a melted state, in the course of continuous bulk polymerization of a mixture of 20 to 100% component (A), 0 to 60% component (B), 0 to 80% component (C), and 0 to 60% component (D).

LEGAL STATUS

LEGAL STATUS	
[Date of request for examination]	23.02.1998
[Date of sending the examiner's decision of rejection]	16.05.2000
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	3158901
[Date of registration]	16.02.2001
[Number of appeal against examiner's decision of rejection]	
[Date of requesting appeal against examiner's decision of rejection]	
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3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim(s)]
 [Claim 1] 20 - 100 % of the weight of aromatic vinyl system monomers, 0 - 60 % of the weight of vinylcyanide system monomers, (Meta) an acrylic ester -- a system -- a monomer -- zero - 80 -- % of the weight -- and -- these -- copolymerization -- being possible -- others -- a vinyl -- a system -- a monomer -- zero - 60 -- % of the weight -- from -- becoming -- a monomer -- mixture -- continuation -- a bulk polymerization -- carrying out -- making -- a process -- inside -- melting -- a state -- a copolymer -- (-- A --) -- ten - 95 -- a weight -- the section -- so much -- Under existence of the rubber-like polymer 5 - 80 weight sections, 10 - 100 % of the weight of aromatic vinyl system monomers, 0 - 50 % of the weight of vinylcyanide system monomers And (meta) the slot which passes a liquefied object for the slurry or water cake obtained from the latex which carried out the graft polymerization of the monomer mixture 95 which consists of 0 - 80 % of the weight of acrylic-ester system monomers, and 0 - 60 % of the weight of vinyl system monomers of others in which these and copolymerization are possible - the 20 weight sections, a hole -- or -- a gap -- and -- the Bento -- having -- extrusion -- equipment -- supplying -- dehydration -- dryness -- carrying out -- having made -- a graft copolymer -- (-- B --) -- 90 - five -- a weight -- the section -- continuous -- adding -- mixing -- things -- the feature -- ** -- carrying out -- shock resistance -- a resin -- a constituent -- manufacture -- a method .

[Claim 2] The manufacture method of a shock-proof resin constituent according to claim 1 that the amount of residual monomers adds a graft copolymer (B) continuously to 10 or less % of the weight of a copolymer (A).

adds a graft copolymer (B) continuously to 10 or less % of the weight of a copolymer (A) -- the manufacture method of in process or [Claim 3] the ** monomer of the continuation bulk polymerization of a copolymer (A) -- the manufacture method of in process or the shock-proof resin constituent according to claim 1 which adds a graft copolymer (B) to the copolymer (A) from which the amount of residual monomers became 10 or less % of the weight henceforth [a ** monomer process]

[Claim 4] The manufacture method of the shock-proof resin constituent according to claim 1 which adds a graft copolymer (B) in the state of half-melting or melting

[Claim 5] The manufacture method of a shock-proof resin constituent according to claim 1 that the rubber-like polymer of a graft copolymer (B) is diene system rubber.

[Claim 6] The manufacture method of the shock-proof resin constituent according to claim 1 which a copolymer (A) is a styrene acrylonitrile copolymer and is the graft copolymer which the graft copolymer (B) made carry out the graft copolymerization of the

[Claim 7] The manufacture method of the shock-proof resin constituent according to claim 1 which the ** monomer process of styrene-acrylonitrile to a rubber-like polymer.

[Claim 7] The manufacture method of the shock proof resin combination, in which the continuation bulk polymerization of a copolymer (A) is the extruder of a monopodium with a vent, or two shafts, and is the extruder of the monopodium or two shafts which the continuation addition equipment of a graft copolymer (B) connected to the ** monomer extruder of a copolymer (A).

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] this invention relates to the manufacture method of a shock-proof resin constituent. It is related with the manufacture method of a shock-proof resin constituent of having excelled in the physical-properties balance of a color tone and shock resistance, and rigidity etc. in more detail.

[0002]

[Description of the Prior Art] The shock-proof resin which blended the rubber component represented by ABS and the high impact polystyrene is a resin excellent in the balance of various physical properties and fabricating-operation nature, and is used for wide range uses, such as autoparts, electrical machinery and apparatus parts, and business-machine machine parts. These rubber component combination shock resistance resins needed to make graft polymerization the rubber component, in order to make sufficient mechanical physical properties discover, and they were conventionally performed by emulsification graft polymerization as the manufacture method. However, since an emulsion-polymerization method has many auxiliary materials, a process becomes [many / cost quantity], and it has problems, like moreover waste water treatment is needed. Therefore, the method of carrying out the melting blend of the high rubber content polymer which carried out the emulsification graft polymerization of the trouble of this emulsion polymerization to the ** sake few, and the polymer obtained according to the suspension polymerization which does not contain rubber has been developed (edited by Society of Polymer Science, Japan "ABS plastics"). Furthermore, the process which carries out the continuation bulk polymerization of the shock-proof resin which contained direct rubber in recent years is also put in practical use (for example, JP,47-14136,B, JP,49-26711,B, chemical engineering 53(6)423-426, etc. (1989)).

[0003]

[Problem(s) to be Solved by the Invention] However, although the method of carrying out a melting blend has the advantage which can control physical properties comparatively smoothly after obtaining respectively the polymer obtained according to the continuation bulk-polymerization method or suspension polymerization which does not contain the high rubber content polymer which carried out emulsification graft polymerization, and rubber as a isolation polymer, in order to receive the heat history further at the time of a melting blend, a color tone is not enough, and there is a fault that the physical-properties balance of shock resistance and rigidity is not enough. Although a process and an auxiliary material are most excellent in the point that waste water treatment is unnecessary, few, control of the graft polymerization reaction in a bulk polymerization is difficult for the method of on the other hand manufacturing the shock-proof resin containing direct rubber by the continuation bulk-polymerization method, and in order that a rubber component may receive more heat histories, it has the fault that a color tone is not good. Furthermore, it cannot necessarily be satisfied with physical-properties sides, such as shock resistance. Moreover, it also has the fault that the trouble on the manufacture top and quality of the degradation object of rubber piling up in equipment if a rubber component increases, and it exfoliating arises.

[0004]

[Means for Solving the Problem] This invention persons resulted in this invention, as a result of considering wholeheartedly the manufacture method of a shock-proof resin constituent excellent in the physical-properties balance of a color tone and shock resistance, and rigidity etc. this invention Namely, 20 - 100 % of the weight of aromatic vinyl system monomers, 0 - 60 % of the weight of vinylcyanide system monomers, (Meta) an acrylic ester -- a system -- a monomer -- zero - 80 -- % of the weight -- and -- these -- copolymerization -- being possible -- others -- a vinyl -- a system -- a monomer -- zero - 60 -- % of the weight -- from -- becoming -- a monomer -- mixture -- continuation -- a bulk polymerization -- carrying out -- making -- a process -- inside -- melting -- a state -- a copolymer -- (A) -- ten - 95 -- a weight -- the section -- so much -- Under existence of the rubber-like polymer 5 - 80 weight sections, 10 - 100 % of the weight of aromatic vinyl system monomers, 0 - 50 % of the weight of vinylcyanide system monomers And (meta) the slot which passes a liquefied object for the slurry or water cake obtained from the latex which carried out the graft polymerization of the monomer mixture 95 which consists of 0 - 80 % of the weight of acrylic-ester system monomers, and 0 - 60 % of the weight of vinyl system monomers of others in which these and copolymerization are possible - the 20 weight sections, a hole -- or -- the Bento -- having -- extrusion -- equipment -- supplying -- dehydration -- dryness -- carrying out -- having made -- a graft copolymer -- (B) -- 90 - five -- a weight -- the section -- continuous -- adding -- mixing -- things -- the feature -- ** -- carrying out -- shock resistance -- a resin -- a constituent -- manufacture -- a method -- providing -- a thing -- it is .

[0005] The aromatic vinyl system monomer which constitutes the copolymer (A) and graft copolymer (B) which are used by this invention is an aromatic compound which has the double bond in which a polymerization is possible, and styrene, an alpha methyl styrene, p-methyl styrene, vinyltoluene, propyl styrene, butyl styrene, cyclohexyl styrene, etc. are mentioned as an example. These aromatic vinyl system monomers are used with one sort or two sorts or more of mixture. Styrene and an alpha methyl styrene are especially used preferably among these aromatic vinyls system monomers. The vinylcyanide system monomer which constitutes the copolymer (A) and graft copolymer (B) which are used by this invention is a compound which reaches the double bond in which a polymerization is possible, and has a cyano group, and acrylonitrile, a methacrylonitrile, etc. are mentioned as an example. These vinylcyanide system monomers are used with one sort or two sorts or more of mixture. Acrylonitrile is especially used preferably among these vinylcyanides system monomers.

[0006] As an example of the acrylic-ester (meta) system monomer which constitutes the copolymer (A) and graft copolymer (B) which are used by this invention, a methyl methacrylate, an ethyl methacrylate, a methacrylic-acid propyl, methacrylic-acid butyl, a glycidyl methacrylate, methacrylic-acid hydroxyethyl, a methyl acrylate, an ethyl acrylate, an acrylic-acid propyl, a butyl acrylate, etc. are mentioned. These (meta) acrylic-ester system monomers are used with one sort or two sorts or more of mixture. A methyl methacrylate is especially used preferably among these (meta) acrylic-esters system monomers. With the vinyl system monomer of others which constitute the copolymer (A) and graft copolymer (B) which are used by this invention, for example, N-phenyl maleimide, N-cyclohexyl maleimide, methylation N-phenyl maleimide, a maleic anhydride, an acrylic acid, a methacrylic acid, etc. are mentioned. N-phenyl maleimide is used especially preferably.

[0007] With the rubber-like polymer which constitutes the graft copolymer (B) used by this invention They are diene system rubber, acrylic rubber, ethylene system rubber, etc. as an example A polybutadiene, poly (styrene butadiene rubber), poly (butadiene-acrylonitrile), A polyisoprene, poly (butadiene-butyl acrylate), poly (butadiene-methyl acrylate), Poly (butadiene-methyl methacrylate), poly (butadiene-ethyl acrylate), Ethylene-propylene rubber, ethylene-propylene diene rubber, poly (ethylene-isobutylene), poly (ethylene-methyl acrylate), poly (ethylene-methyl acrylate), etc. are mentioned. These rubber-like polymers are used with one sort or two sorts or more of mixture. A polybutadiene, poly (styrene butadiene rubber), poly (butadiene-acrylonitrile), and ethylene-propylene rubber are especially used preferably among these rubber-like polymers.

[0008] As a desirable example of the copolymer (A) used by this invention, polystyrene, a styrene acrylonitrile copolymer, a styrene-N-phenyl maleimide copolymer, a styrene-acrylonitrile-methyl-methacrylate copolymer, and a styrene-methyl-methacrylate copolymer are mentioned, and a styrene acrylonitrile copolymer is used especially preferably.

[0009] As a desirable example of the graft copolymer (B) used by this invention, the styrene graft polymer of a polybutadiene, A poly (styrene butadiene rubber) styrene graft polymer, the styrene-acrylonitrile graft copolymer of a polybutadiene, A poly (styrene butadiene rubber) styrene-acrylonitrile graft copolymer, A poly (butadiene-acrylonitrile) styrene-acrylonitrile graft copolymer, The styrene-acrylonitrile-methyl-methacrylate graft copolymer of a polybutadiene, a poly (ethylene-propylene) styrene-acrylonitrile graft copolymer, etc. are mentioned. Let especially the operating rates of each monomer of a copolymer (A) be 20 - 100 % of the weight of aromatic vinyl system monomers, 0 - 60 % of the weight of vinylcyanide system monomers, 0 - 80 % of the weight of acrylic-ester (meta) system monomers, and 0 - 60 % of the weight of vinyl system monomers of others in which these and copolymerization are possible especially from a viewpoint of the mechanical strength of the resin constituent obtained, a color tone, and a moldability. It is desirable 30 - 100 % of the weight of aromatic vinyl system monomers, 0 - 50 % of the weight of vinylcyanide system monomers, 0 - 70 % of the weight of acrylic-ester (meta) system monomers, and 0 - 50 % of the weight of vinyl system monomers of others in which these and copolymerization are possible, and is more desirable 60 - 100 % of the weight of aromatic vinyl system monomers, 10 - 40 % of the weight of vinylcyanide system monomers, 0 - 60 % of the weight of acrylic-ester (meta) system monomers, and 0 - 40 % of the weight of vinyl system monomers of others in which

[0010] There is no limit in the continuation bulk-polymerization method in the process to which the continuation bulk polymerization of the monomer mixture which consists of 20 - 100 % of the weight of processes in the first half of this invention, i.e., an aromatic vinyl system monomer, 0 - 80 % of the weight of 0 - 60 % of the weight (meta) acrylic-ester system monomers of vinylcyanide system monomers, and 0 - 60 % of the weight of vinyl system monomers of others in which these and copolymerization are possible is carried out, and any continuation bulk-polymerization methods can adopt it as it. For example, after carrying out a polymerization by the polymerization tank, the method of carrying out a ** monomer (deaeration) etc. is learned. the polymerization tank of a mixed type which has various kinds of impellers, for example, paddle wings, a turbine blade, propeller wings, bloomers gin wings, multi-stage wings, support wings, the Max blend wings, double helical wings, etc. as a polymerization tank, or various kinds of columns -- the reactor of a type etc. can be used A multi-pipe reactor, a kneader formula reactor, a twin screw extruder, etc. can also be used as a polymerization reactor further again (for example, the assessment 10 "assessment of high impact polystyrene":Society of Polymer Science, Japan of a macromolecule manufacture process, January 26, 1989, etc.). These polymerization tanks (reactor) are used above one set (tub) or two set (tub), and even if it combines two or more kinds of reactors if needed, it can be used.

[0011] Next a ** monomer process is usually presented with the reaction mixture of the copolymer (A) which carried out the polymerization with these polymerization tanks or reactors, and the volatile component of a monomer and others is removed. The way the extruder of one shaft which has a vent, or two shafts removes a volatile component from a vent hole by the bottom ordinary pressure of heating, or reduced pressure as the method of a ** monomer, The way the evaporator which carries out the viscous of the plate fin type heaters, such as a centrifugal type, to a drum removes a volatile component, Although there are remaining heat, a method of foaming, carrying out a flash plate to a vacuum tub, and removing a volatile component, etc. using the method and multibular heat exchanger which remove a volatile component by film evaporators, such as a centrifugal type, and

any method can be used, the extruder of one shaft which has especially a vent, or two shafts is used preferably. [0012] The continuation bulk polymerization of a copolymer (A) can also use [also carrying out thermal polymerization, without using an initiator, also carrying out an initiator polymerization using an initiator, and] thermal polymerization and an initiator polymerization together further. A peroxide or an azo system compound is used as an initiator.

[0013] As an example of a peroxide, benzoyl peroxide, a cumene hydroperoxide, Dicumyl peroxide, diisopropylbenzene hydroperoxide, t-butyl hydroperoxide, t-butyl cumyl peroxide, Tert-butyl peroxide acetate, t-butyl par oxybenzoate, Tert-butyl peroxide isopropyl cull BONETO, G t-butyl par OKIIDO, t-butyl par OKUTETO, 1, and 1-screw (tert-butyl peroxide) 3 and 3, 5-trimethyl cyclohexane, 1, and 1-screw (tert-butyl peroxide) cyclohexane, t-butylperoxy2-ethylhexanoate, etc. are mentioned. The cumene hydroperoxide and 1, and 1-screws (tert-butyl peroxide) 3 and 3 and a 5-trimethyl cyclohexane are used especially preferably. As an example of an azo system compound, an azobisisobutyronitril, azobis (2,4-dimethylvaleronitrile), 2-phenylazo-2, 4-dimethyl-4-methoxy valeronitrile, 2-cyano-2-propyl azo formamide, 1, and 1'-azobis cyclohexane-1-carbo nitril, Azobis (4-methoxy-2,4-dimethylvaleronitrile), a dimethyl 2, 2'-azobisisobutyrate, A 1-t-butyl azo-1-cyano cyclohexane, 2-t-butyl azo-2-cyano butane, 2-t-butyl azo - A 2-cyano-4-methoxy-4-methyl pentane etc. is mentioned. When using these initiators, one sort or two sorts or more are used, using together. 1 and 1'-azobis cyclohexane-1-carbo nitril is used especially preferably.

[0014] It is also possible to use chain transfer agents, such as a mercaptan and a terpene, for the purpose of polymerization-degree regulation of the copolymer (A) used by this invention, and n octyl mercaptan, t-dodecyl mercaptan, n-dodecyl mercaptan, n-tetradecyl mercaptan, n-octadecyl mercaptan, a terpinolene, etc. are mentioned as the example. When using these chain transfer agents, one sort or two sorts or more are used, using together. N octyl mercaptan, t-dodecyl mercaptan, and n-dodecyl mercaptan are used preferably also especially in inside. Although the copolymer (A) used by this invention is manufactured by the continuation bulk-polymerization method, it is possible also for carrying out a polymerization using a little (for example, 20% or less) solvent, and is contained in the range of this invention.

[0015] The graft copolymer (B) which is another [which is used by this invention] constituent In the rubber-like polymer 5 - 80 weight sections, 10 - 100 % of the weight of aromatic vinyl system monomers, Although it is the copolymer which carries out the graft polymerization reaction of the monomer mixture 95 which consists of 0 - 50 % of the weight of vinylcyanide system monomers, 0 - 80 % of the weight of acrylic-ester (meta) system monomers, and 0 - 60 % of the weight of vinyl system monomers of others in which these and copolymerization are possible - the 20 weight sections What was obtained as mixture with the copolymer in which the whole quantity does not need to carry out a graft, and which has not usually carried out a graft is used. Although there is no limit in the rate of a graft of a graft copolymer (B), 10 - 100% of the weight of a thing is used more preferably 5 to 150%. The rate of a graft here is computed by the following formula.

Rate (% of the weight) = (graft branch weight / amount of rubber-like polymerization weights) x of graft 100. [0016] The rates of the rubber-like polymer in a graft copolymer (B) are the mechanical strength of the resin constituent obtained, a color tone, and 5 - 80 weight section from a viewpoint of a moldability, and are 20 - 70 weight section more preferably. The operating rate of each monomer other than the rubber-like polymer of a graft copolymer (B) 10 - 100 % of the weight of aromatic vinyl system monomers, 0 - 50 % of the weight of vinylcyanide system monomers, They are 0 - 80 % of the weight of acrylic-ester system monomers, and 0 - 60 % of the weight of vinyl system monomers of others in which these and copolymerization are possible. (Meta) More preferably 60 - 100 % of the weight of (1) aromatic vinyl system monomers, 10 - 40 % of the weight of vinylcyanide system monomers (meta), 0 % of the weight of acrylic-ester system monomers, Or they are 20 - 60 % of the weight of (2) aromatic vinyl system monomers, 0 - 30 % of the weight of vinylcyanide system monomers (meta), and 40 - 80 % of the weight of acrylic-ester system monomers.

[0017] The manufacture method of a graft copolymer (B) adopts an emulsion polymerization. Usually, an emulsion polymerization carries out the emulsification graft polymerization of the monomer mixture to rubber-like polymer latex's existence-ization. Although there is especially no limit in the emulsifier used for this emulsification graft polymerization and various kinds of surfactants can be used, anion system surfactants, such as a carboxylate type, a sulfate salt type, and a sulfonate type, are used especially preferably. As an example of such an emulsifier, capryl lactam acid chloride, a caprate, Lauryl acid chloride, misty phosphate, a palmitate, a stearate, An oleate, the linoleate, a linolenic-acid salt, rosin acid chloride, a behenic acid salt, A castor-oil sulfate salt, a lauryl alcohol sulfate salt, other higher-alcohol sulfate salts, The dodecylbenzenesulfonic acid salt, alkylnaphthalenesulfonate, Alkyl diphenyl-ether disulfon acid chloride, a naphthalene sulfonate condensate, The dialkyl sulfo succinate, a polyoxyethylene lauryl sulfate, a polyoxyethylene-alkyl-ether sulfate, a polyoxyethylene-alkyl-phenyl-ether sulfate, etc. are mentioned. Salts here are an alkali-metal salt, an ammonium salt, etc., and potassium salt, sodium salt, lithium salt, etc. are mentioned as an example of an alkali-metal salt. They are used for these emulsifiers for one sort or two sorts or more, using together. Moreover, the initiator and chain transfer agent which were raised with these emulsification graft polymerization by manufacture of the aforementioned copolymer (A) as an usable initiator and a chain transfer agent are mentioned, and, as for an initiator, a redox system is also used.

[0018] Next the graft copolymer (B) manufactured by emulsification graft polymerization adds a coagulant, solidifies a latex, and collects graft copolymers (B). An acid or a water-soluble salt is used as a coagulant, and a sulfuric acid, a hydrochloric acid, a phosphoric acid, an acetic acid, a calcium chloride, a magnesium chloride, barium chloride, an aluminum chloride, magnesium sulfate, an aluminum sulfate, an aluminum ammonium sulfate, a potassium aluminum sulfate, an aluminium sodium sulfate, etc. are mentioned as the example. These coagulants are used with one sort or two sorts or more of mixture. The extrusion equipment which has the slot, the hole or gap, and Bento which pass a liquefied object is supplied, the graft copolymer (B) of the shape of the solidified slurry or a water cake is dehydrated, and it is made to dry. This extrusion equipment dehydrated and dried consists of

a screw, a cylinder, and a screw mechanical component, and, as for a cylinder, it is desirable to have heating and refrigeration capacity. And although a cylinder passes a liquefied object for the first portion (supply side), most solid contents may have the slot, hole, or gap which is not passed, and may have one or more Bento in the section (discharge side) in the second half, and a monopodium extruder or a biaxial extruder is sufficient as them. The graft copolymer (B) of the shape of a slurry or a water cake is supplied to this extrusion equipment, after compressing by rotation of a screw in the low-temperature field (first portion) of a cylinder and making a great portion of water discharge from the slot, hole, or gap of the cylinder first portion (supply side), residual moisture and residual volatile matter are removed from the Bento in the heating region of the section (discharge side) in the second half of a cylinder, and a copolymer (A) is supplied more nearly continuously than a cylinder. Also with an ordinary pressure, the Bento may be pulled to reduced pressure and may use together an ordinary pressure and reduced pressure by two more or more Bento.

[0019] In this invention, after adding a graft copolymer (B) continuously to the copolymer (A) in the melting state in a bulk-polymerization process, it is necessary to mix and the resin constituent which was [shock resistance / a color tone,] excellent for the first time with it is obtained. Moreover, it is necessary to add 90 - 5 weight section for a graft copolymer (B) continuously in the copolymer (A) 10 in a melting state - 95 weight sections, and in that case, more preferably, a graft copolymer (B) is mixed in them, after adding 70 - 5 weight section continuously in a copolymer (A) 30 - 95 weight sections. addition of the graft copolymer in this case (B) -- the ** monomer of the bulk-polymerization process of a copolymer (A) -- since a color tone, shock resistance, etc. which a rubber component does not deteriorate by the heat history during subsequent ** monomer operation, but are the feature of this invention become still better in process if it carries out in that the amount of residual monomers became 5% or less more preferably 10% or less henceforth [a ** monomer process], it is desirable. Moreover, in this invention, the mixture after adding a graft copolymer (B) continuously to a copolymer (A) is desirable, in order for carrying out melting mixture to make physical properties, such as shock resistance, fully discover. Even if it performs this melting mixture at the time of addition mixture, you may perform it after mixture isolation (at for example, the time of melting fabrication).

[0020] It is possible for there to be especially no limit in the continuation addition method of a graft copolymer (B), and to add by arbitrary methods. Usually, although various kinds of feeders, for example, a belt formula feeder, a screw-type feeder, a single screw extruder, a twin screw extruder, etc. are used, a single screw extruder and a twin screw extruder are used especially preferably. As for these continuation addition equipment, what can carry out a fixed quantity is desirable. Moreover, the mixed state becomes good and is desirable [equipment], when continuation addition equipment has heating apparatus and a graft copolymer (B) is added in the state of half-melting or melting. The extruder which has heating apparatus can be used for this purpose.

[0021] In this invention, it is also possible to blend reinforcing materials, such as lubricant, such as weathering agents, such as various kinds of antioxidants, such as a phenol system, the Lynn system, and a sulfur system, an ultraviolet ray absorbent, and a light stabilizer, an antistatic agent, ethylene-bis-stearylamide, and metal soap, a plasticizer, a coloring agent, a bulking agent, a glass fiber, and a carbon fiber, a flame retarder, etc. if needed further.

[0022] [Example] Although an example is given below and this invention is explained further in full detail, this invention is not limited to these examples. In addition, % and the section which were used by this example show weight % and the weight section respectively. Moreover, YI value of a pellet measured the yellow index (YI value) using the color difference meter made from SUGA Testing machine. Moreover, Izod impactive strength is ASTMD256 and tensile strength is ASTM. It measured according to 638.

[0023] The example 1 (manufacturing method of a graft copolymer) of reference
The polybutadiene latex (0.3 micrometer [of rubber particle systems], 85% of gel contents) 50 section (solid-content conversion), the pure water 200 section, the sodium formaldehyde sulfoxylate 0.4 section, the ethylenediaminetetraacetic acid sodium 0.1 section, a ferrous sulfate (0.01 sections), and the sodium phosphate 0.1 section. It taught the reaction container, the ** tone was carried out to 65 degrees C after the nitrogen purge, and continuation dropping of the mixture of the bottom styrene of churning 35 section, the acrylonitrile 15 section, and the n-dodecyl-mercaptopan 0.3 section was carried out over 4 hours. Simultaneously, continuation dropping was carried out over 5 hours, the mixture of the cumene hydroperoxide 0.25 section, the lauric-acid sodium 2.5 section which is an emulsifier, and the pure water 25 section was held after the dropping end in parallel, for further 1 hour, and the polymerization was terminated. the latex which ended the polymerization -- 1.5% sulfuric acid -- solidifying -- subsequently -- alkali -- neutralization, washing, and centrifugal separation -- it dried and the graft copolymer on powder (B-1) was adjusted. Extracting the obtained graft copolymer (B-1) by MEK, the rate of a graft was 45%.

[0024] The example 2 (manufacturing method of a graft copolymer) of reference
The graft copolymer (B-1) latex was manufactured like the example 1 of reference. After solidifying this latex with a sulfuric acid 1.5%, it neutralized with alkali and the graft copolymer slurry (B-2) was prepared.

[0025] The examples 3-11 (manufacturing method of a graft copolymer) of reference
The graft polymer cake (-3-11) which has the composition which carried out the polymerization of the mixture with styrene and other vinyl monomers to the bottom of existence of the various rubber-like polymers shown in Table 3 like the example 1 of reference, and was shown in Table 3 was manufactured. In addition, for the styrene / pig JIEN copolymer rubber which consists of styrene 25% and pig JIEN 75%, and NBR, with PBD in Table 1, the acrylonitrile / pig JIEN copolymer rubber which consists of acrylonitrile 25% and pig JIEN 75%, and EPDM are [having used it in the example 1 of reference, the same poly pig JIENGOMU, and SBR] propylene / ethylene [of the iodine number 23 and Mooney viscosity 60] /

5 ethylenic-2-NORIURONEN ternary polymerization object rubber (ethylene / propylene It expresses.

[0026] Specification shown in example 1 table 1. From the nose of cam of two tubs which it has, a preheater, a ** monomer machine, and a ** monomer machine, using the continuous system bulk-polymerization equipment which consists of a biaxial extruder type feeder linked to the tandem which has heating apparatus, the monomer mixture which consists of the styrene 70 section, the acrylonitrile 30 section, and the n-octyl-mercaptan 0.15 section was supplied to 1/3 merit's barrel section continuously [kg / 150 //o'clock] to the 1st polymerization tank, and carried out the continuation bulk polymerization to it. The conversion of the 1st polymerization tank is for 58 - 61%, and the polymer of the 2nd polymerization tank appearance was controlled and operated among 90 - 91%. Polymerization-reaction mixture is TORI (nonylphenyl) phosphite 0.15kg/o'clock which is the stabilizer of the t-butylhydroxytoluene 0.15kg [which is the stabilizer of a phenol system from a biaxial extruder type feeder at the styrene/acrylonitrile copolymer to which reduced pressure evaporation recovery of the unreacted monomer was carried out from the vent-port with the single-screw-extruder type ** monomer machine, it saw from the nose of cam of a ** monomer machine in one third of places, and the upper conversion went up to 99% or more]/o'clock, and Lynn system. After having dehydrated the graft copolymer (B-1) manufactured in the example 1 of reference by the single screw extruder equipped with two Bento, supplying by 65kg/o'clock in speed in the state of half-melting and carrying out melting kneading with styrene/acrylonitrile copolymer with a ** monomer machine, it was made to breathe out in the shape of a strand, and the styrene-resin constituent pellet was obtained by the cutter. YI value of the obtained styrene-resin constituent was as having been shown in Table 4. Moreover, the piece of injection molding of the obtained styrene-resin constituent was fabricated, and the result which measured physical properties was shown in Table 4. The styrene-resin constituent manufactured by the method of this invention was excellent in a color tone and physical properties as I understood from Table 4.

[0027] Except having changed to the graft copolymer cake (B-1) of example 2 example 1, having dehydrated by the single screw extruder equipped with the gap and two Bento which pass water for the graft copolymer slurry (B-2) manufactured in the example 2 of reference, and having supplied by 65kg/o'clock in speed in the state of half-melting, it carried out like the example 1 and the styrene-resin constituent pellet was obtained. YI value of the obtained styrene-resin constituent was as having been shown in Table 4. Moreover, the piece of injection molding of the obtained styrene-resin constituent was fabricated, and the result which measured physical properties was shown in Table 4. The styrene-resin constituent manufactured by the method of this invention as shown in Table 4 was excellent in a color tone and physical properties.

as shown in Table 4 was excellent in a color tone and physical properties. [0028] They are a continuation bulk polymerization / ** monomer machine about monomer mixture given in Table 4 like an example 1 except supplying at the speed which showed the graft copolymer cake (B-3-11) manufactured in the examples 3-11 of reference from the single screw extruder equipped with two Bento heated example 3-11 in Table 4 in the state of half-melting. The unreacted monomer was made to breathe out in the shape of a strand with reduced pressure evaporation recovery and a ** monomer machine after carrying out melting kneading of a styrene system copolymer (A) and the graft copolymer (B), and the styrene-resin constituent pellet was obtained. The physical-properties measurement result of the test piece obtained by carrying out injection molding of YI value of a styrene-resin constituent and the resin constituent which were obtained was shown in Table 4. The styrene-resin constituent manufactured by the method of this invention as shown in Table 4 was excellent in a color tone and physical properties.

[0029] Using the same continuous system bulk-polymerization equipment as example 12 example 1, with the speed of 150kg/o'clock, the monomer mixture which consists of the styrene 100 section and the t-butyl-mercaptopan 0.15 section was continuously supplied to the 1st polymerization tank, and carried out the continuation polymerization to it. The conversion of the 1st polymerization tank appearance is 67 - 70%, and the conversion of the 2nd polymerization tank appearance was controlled and operated among 90 - 91%. Polymerization-reaction mixture is the biaxial extruder type ** monomer machine after preheating by the single-screw-extruder type preheater like the example 1. To the styrene polymer to which vacuum distillation recovery of the unreacted monomer was carried out from the vent-port, it saw from the nose of cam of a ** monomer machine in one third of places, and the upper conversion went up to 99% or more. The graft copolymer cake (B-4) manufactured in the example 4 of reference with t-butylhydroxytoluene 0.15kg/o'clock from the heated biaxial extruder type feeder is dehydrated by the single screw extruder equipped with two Bento. After supplying by 65kg/o'clock in speed in the state of half-melting and carrying out melting kneading with a styrene polymer with a ** monomer machine, it breathed out in the shape of a strand, and the styrene-resin constituent pellet was obtained by the cutter. The physical-properties measurement result of the test piece obtained by carrying out injection molding of YI value of a styrene-resin constituent and this resin constituent which were obtained was shown in Table 4. The styrene-resin constituent manufactured by the method of this invention as shown in Table 4 was excellent in a color tone and physical properties.

[0030] Specification of example 13 table 2. The monomer mixture which consists of the styrene 67 section, the acrylonitrile 33 section, the n-octyl-mercaptan 0.18 section, and the t-butyl peroxide 0.01 section was continuously supplied to the polymerization tank, and was made it to carry out a continuation polymerization to 1/3 merit's barrel section by 150kg/o'clock in speed using the continuous system bulk-polymerization equipment which consists of a biaxial extrusion type feeder which has the heating apparatus linked to the tandem from the nose of cam of one tub which it has, a preheater, a ** monomer machine, and a ** The conversion of polymerization tank appearance was controlled among 74 - 76%, and it operated. Polymerization-reaction mixture is the biaxial extruder type ** monomer machine after preheating by the single-screw-extruder type preheater. To the styrene/acrylonitrile copolymer to which vacuum distillation recovery of the unreacted monomer was carried out from the vent-port, it saw from the nose of cam of a ** monomer machine in one third of places, and the upper conversion went up to 99% or more With TORI (nonylphenyl) phosphite 0.15kg/the o'clock which is the stabilizer of the t-butylhydroxytoluene 0.15kg

which is a phenol system stabilizer 1/0'clock, and Lynn system The graft copolymer cake (B-6) manufactured in the example 6 of reference was dehydrated by the single screw extruder equipped with two Bento, and it supplied by 65kg/0'clock in speed in the state of half-melting, and it pelletized, after carrying out melting kneading with styrene/acrylonitrile copolymer with a ** monomer machine. The physical-properties measurement result of the test piece obtained by carrying out injection molding of YI value and this resin constituent of a styrene-resin constituent pellet which were obtained was shown in Table 4. As shown in Table 4, the styrene-resin constituent manufactured by the method of this invention was excellent also with a color tone and physical properties.

[0031] Using the same continuous system bulk-polymerization equipment as example 14 example 11, by 150kg/0'clock in speed, the monomer / solvent mixture which consists of the styrene 49 section, the acrylonitrile 21 section, the N-phenyl maleimide 30 section, the toluene 10 section, the n-octyl-mercaptopan 0.18 section, and the t-butyl peroxide 0.01 section were continuously supplied to the polymerization tank, and carried out the continuation polymerization to it. The conversion of polymerization tank appearance was controlled among 74 - 76%, and it operated. Polymerization-reaction mixture is the biaxial extruder type ** monomer machine after preheating by the single-screw-extruder type preheater. Vacuum distillation recovery of an unreacted monomer and the toluene is carried out from a vent-port. To the styrene / acrylonitrile / N-phenyl maleimide copolymer to which it saw in one third of places, and the upper conversion went up from the nose of cam of a ** monomer machine to 99% or more, with t-butylhydroxytoluene 0.15kg/0'clock, and TORI (nonylphenyl) phosphite 0.15kg/0'clock The graft copolymer cake (B-1) manufactured in the example 1 of reference is dehydrated by the single screw extruder equipped with two Bento. It supplied by 65kg/0'clock in speed in the state of half-melting, and it pelletized, after carrying out melting kneading with styrene / acrylonitrile / N-phenyl maleimide copolymer with a ** monomer machine. The physical-properties measurement result of the test piece obtained by carrying out injection molding of YI value and this resin constituent of an imido ** resin pellet which were obtained was shown in Table 4. As shown in Table 4, the styrene-resin constituent manufactured by the method of this invention was excellent also with a color tone and physical properties.

[0032] Using the continuous system bulk-polymerization equipment which consists of polymerization tank 2 tub which has the specification of example of comparison 1 table 1, a preheater, and a ** monomer machine, by 150kg/0'clock in speed, the monomer mixture which consists of the styrene 70 section, the acrylonitrile 30 section, and the n-octyl-mercaptopan 0.18 section was continuously supplied to the 1st polymerization tank, and carried out the continuation polymerization to it. The conversion of the 1st polymerization tank appearance is for 58 - 61%, and the conversion of the 2nd polymerization tank appearance was controlled and operated among 90 - 91%. Polymerization-reaction mixture carried out vacuum distillation recovery of the unreacted monomer from the vent-port with the biaxial extruder type ** monomer machine, made apparent conversion 99% or more, breathed it out in the shape of a strand, and was pelletized by the cutter. The graft copolymer cake (B-1) manufactured in the obtained styrene / acrylonitrile-copolymer pellet, and the example 1 of reference was dried, after carrying out dryblend at a rate shown in Table 4, it melting-kneaded, / extruded, it pelletized and the styrene-resin constituent pellet was obtained. The property measurement result of the test piece obtained by carrying out injection molding of YI value and this resin constituent of a styrene-resin constituent pellet which were obtained was shown in Table 4. The styrene-resin constituent clearly manufactured in this example of comparison compared with the example was what is inferior in a color tone (pellet YI).

[0033] The graft polymer cake (B-1) which manufactured the monomer mixture which consists of the example of comparison 2 styrene 70 section, the acrylonitrile 30 section, and the t-dodecyl-mercaptopan 0.18 section in the styrene / acrylonitrile-copolymer bead which carried out the polymerization and was obtained by dehydrating and drying by the suspension polymerization, and the example 1 of reference was dried, after carrying out dryblend at a rate shown in Table 4, melting kneading / extrusion PERETAISU was carried out, and the styrene-resin constituent pellet was obtained. The physical-properties measurement result of the test piece obtained by carrying out injection molding of YI value and this resin constituent of a styrene-resin constituent pellet which were obtained was shown in Table 4. The styrene-resin constituent clearly manufactured in this example of comparison compared with the example was what is inferior in a color tone (pellet I).

[0034] Using the same continuous system bulk-polymerization equipment as example of comparison 3 example 1, by 150kg/0'clock in speed, the monomer / solvent mixture which consists of the styrene 49 section, the acrylonitrile 21 section, the N-phenyl maleimide 30 section, the toluene 10 section, the n-octyl-mercaptopan 0.18 section, and the t-butyl peroxide 0.01 section were continuously supplied to the polymerization tank, and carried out the continuation polymerization to it. The conversion of polymerization tank appearance was controlled among 74 - 76%, and it operated. After preheating polymerization-reaction mixture by the single-screw-extruder type preheater, it carried out vacuum distillation recovery of an unreacted monomer and the toluene from the vent-port with the biaxial extruder type ** monomer machine, made apparent conversion 99% or more, breathed it out in the shape of a strand, and was pelletized by the cutter. The obtained styrene / acrylonitrile / N-phenyl maleimide copolymer pellet, and the graft copolymer cake (B-1) manufactured in the example 1 of reference were dried, after carrying out dryblend at a rate shown in Table 4, it melting-kneaded, / extruded, it pelletized and the imido ** resin constituent pellet was obtained. The physical-properties measurement result of the test piece obtained by carrying out injection molding of YI value and this resin constituent of a styrene-resin constituent pellet which were obtained was shown in Table 4. The imido ** resin constituent clearly manufactured in this example of comparison compared with the example was what is inferior in a color tone (pellet YI).

[0035] The graft copolymer cake (B-1) of example of comparison 4 example 1 was dehydrated by the single screw extruder equipped with two Bento, and the styrene-resin constituent was obtained like the example 1 except having carried out the amount of supply in 5kg/0'clock in the state of half-melting. The physical-properties measurement result of the test piece obtained by

carrying out injection molding of YI value and this styrene-resin constituent of a styrene-resin constituent pellet which were obtained was shown in Table 4. The styrene-resin constituent clearly manufactured in this example of comparison compared with the example was that in which Izod impactive strength is inferior.

[0036] The styrene-resin constituent was obtained like the example 1 except having carried out the amount of supply of 9kg /and a graft copolymer cake (B-1) for the amount of supply of the styrene of example of comparison 5 example 1, and the monomer mixture of acrylonitrile in 92kg/o'clock o'clock. The physical-properties measurement result of the test piece obtained by carrying out injection molding of YI value and this styrene resin constituent of a styrene-resin constituent pellet which were obtained was shown in Table 4. The styrene-resin constituent clearly manufactured in this example of comparison compared with the example was that in which tensile strength is inferior.

[0037]

[Table 1]

	第1重合槽	第2重合槽
機体型式	ヘリカルリボン翼を有する完全混合タイプ	多段式多孔板とかき取り翼を有する栓流タイプ
温度 (°C)	115	上部 130 中部 155 下部 195
圧力 (kg/cm ² G)	0.9	1.2
冷却装置	ジャケットおよび単量体蒸気の蒸発過流用コンデンサーを有する	

[Table 2]

機体型式	ヘリカルリボン翼を有する完全混合タイプ
温度 (°C)	135
圧力 (kg/cm ² G)	0.9
冷却装置	ジャケットおよび単量体蒸気の蒸発過流用コンデンサー

[Table 3]

参考例	グラフト 共重合体 N.O.	ゴム重合体 ゴム種	グラフト共重合体 (重量%)				グラフト率 (重量%)
			ゴム状重合体 PBD	ゴム状重合体 PBD	アクリロニトリル ステレン	メタクリル酸メチル N-フェニルマレイミド	
1	ケーク B-1	PBD	50	35	15		46
2	スラリー B-2	PBD	50	35	15		45
3	ケーク B-3	PBD	45	26	12		58
4	ケーク B-4	PBD	60	40			20
5	ケーク B-5	PBD	45	17	3	35	40
6	ケーク B-6	NBR	60	18	12		30
7	ケーク B-7	SBR	50	37	13		40
8	ケーク B-8	EPDM	40	45	15		45
9	ケーク B-9	PBD	23.3	55	21.7		80
10	ケーク B-10	PBD	10	90			80
11	ケーク B-11	PBD	10	63	27		90

[Table 4]

	共重合体 (A) 組成 スチレン アクリロニトリル N-アセチル マレインイド	供給量 kg/時	種類 グラフト共重合体 供給量 kg/時	混合方法 共重合体 (A) の 組成ノマ-直線	ペント - アイソ- ト- ブリ ー ン	機械的強度 アイソ- ト- ブリ ー ン kgcm/cm	引張り強度 kg/cm ²
実施例1	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-1 6.5 共重合体 (A) の 組成ノマ-直線	2.2 2.5	2.2 2.5	4.00
実施例2	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-2 6.5 同上	2.1 2.3	2.1 2.3	4.30
実施例3	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-3 6.5 同上	2.5 1.5	2.5 1.5	4.50
実施例4	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-4 6.5 同上	2.0 1.2	2.0 1.2	4.50
実施例5	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-5 6.5 同上	2.1 2.0	2.1 2.0	4.20
実施例6	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-6 6.5 同上	2.4 1.5	2.4 1.5	4.50
実施例7	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-7 6.5 同上	2.2 1.9	2.2 1.9	4.10
実施例8	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-8 6.5 同上	2.3 1.4	2.3 1.4	4.30
実施例9	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-9 6.5 同上	1.9 1.1	1.9 1.1	4.50
実施例10	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-10 6.5 同上	1.7 8	1.7 8	4.60
実施例11	70 スチレン	30 アクリロニトリル	1.35 N-アセチル マレインイド	8-11 6.5 同上	1.8 1.0	1.8 1.0	4.50
実施例12	100 アクリロニトリル		1.35 N-アセチル マレインイド	8-4 6.5 同上	2.0 1.5	2.0 1.5	4.40
実施例13	76 アクリロニトリル	33 N-アセチル マレインイド	1.13 1.35 同上	B-6 6.5 同上	2.1 2.0	2.1 2.0	4.10
実施例14	49 アクリロニトリル	21 N-アセチル マレインイド	1.02 1.35 同上	B-1 6.5 ドライブレンド	2.2 1.7	2.2 1.7	4.40
比較例1	70 アクリロニトリル	30 N-アセチル マレインイド		B-1 6.5 ドライブレンド	3.2 2.6	3.2 2.6	4.00
比較例2	70 アクリロニトリル	30 N-アセチル マレインイド		1.35 6.5 ドライブレンド	3.5 2.5	3.5 2.5	4.10
比較例3	49 アクリロニトリル	21 N-アセチル マレインイド		1.02 6.5 ドライブレンド	3.8 1.1	3.8 1.1	4.50
比較例4	70 アクリロニトリル	30 N-アセチル マレインイド		1.35 6.5 共重合体 (A) の 組成ノマ-直線	2.0 3	2.0 3	5.80
比較例5	70 アクリロニトリル	30 N-アセチル マレインイド		8 B-1 6.2 同上	3.8 4.5	3.8 4.5	1.00

[0038]

[Effect of the Invention] When this invention manufactures the resin which does not contain a rubber component by the continuation bulk-polymerization method and a resin is in a melting state in the second half of a ** monomer process, it is the feature to add the graft copolymer containing a rubber component and to mix. Therefore, the resin constituent which was excellent in the color tone and the mechanical strength as examples 1-14 showed is obtained. Moreover, according to the manufacture method of this invention, it becomes possible to decrease waste water treatment, and further, it is few and the reduction of a manufacturing cost also of a manufacturing process is attained.

[Translation done.]